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(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Toshihiro ITO et al.

Application No.: 10/542,200 Confirmation No.: 3457

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For: ENRICHED RICE OR ENRICHED WHEAT Examiner: GEORGE, Patricia Ann

DECLARATION UNDER 37 CFR 1.132

COMMISSIONER FOR PATENTS
P. O. Box 1450
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Sir:

I, Noboru SAKAGUCHI, residing in Mie-ken, Japan, hereby declares and states as follows:

1. That I am a co-inventor of the above-identified application, and thoroughly familiar with the contents of U.S. Application Serial No. 10/542,200 filed on July 15, 2005, entitled ENRICHED RICE OR ENRICHED WHEAT, its prosecution before the United States Patent and Trademark Office and the references cited therein.

2. I am a graduate of The University of Tokushima, Faculty of Engineering and received a master's degree in the year 1979, majoring in applied chemistry.

3. That I have been employed in Taiyo Kagaku Co., Ltd. in the year 1986 and have been assigned to the Research Laboratories.

4. I have been involved in the research and development of nutrition delivery system since 1999.

5. The following experiments were conducted by myself or under my direct supervision and control in order to verify that the present invention is clearly distinguishable from Misaki (U.S. Patent No. 4,765,996).

EXPERIMENTAL METHOD

In the following tests the raw materials described in Misaki were all commercially available products.

Summary of Preparation of Enriched Rice

Enriched Rice 1: The same procedures as in Example 5 of Misaki were carried out to give enriched rice (Enriched Rice 1).

Enriched Rice 2: The same procedures as in Example 5 of Misaki were carried out except that the aqueous suspension containing ferric pyrophosphate described in Example 5 of Misaki was changed to an emulsifying agent-coated iron salt composition A described in Example 1 of the present specification, to give enriched rice (Enriched Rice 2).

Enriched Rice 3: The same procedures as in Example 8 of the present specification were carried out using vitamins and minerals given in Example 5 of Misaki, so as to have final theoretical vitamin content and mineral content of nutrient enriched rice, to

give enriched rice (Enriched Rice 3). Here, the vitamins and the minerals were prepared in the same manner as in Example 3 of the present specification.
Enriched Rice 4: The same procedures as in Example 8 of the present specification were carried out using vitamins and minerals given in Example 5 of Misaki, so as to have final theoretical vitamin content and mineral content of nutrient enriched rice, except that the aqueous suspension containing ferric pyrophosphate described in Example 5 of Misaki was changed to an emulsifying agent-coated iron salt composition A described in Example 1 of the present specification, to give enriched rice (Enriched Rice 4). Here, the vitamins and the minerals were prepared in the same manner as in Example 3 of the present specification.

Detailed Procedures for Preparation of Enriched Rice

Emulsifying Agent-Coated Iron Salt Composition A

An iron solution was prepared by dissolving 130 g of ferric chloride hexahydrate and 3 g of enzymatically decomposed lecithin (SUNLECITHIN L: manufactured by Taiyo Kagaku Co., Ltd.) in 600 g of ion-exchanged water. Also, 200 g of tetrasodium pyrophosphate decahydrate and 17 g of pentaglycerol monomyristate (SUNSOFT A-141E: manufactured by Taiyo Kagaku Co., Ltd.) were dissolved in 5 kg of ion-exchanged water, to prepare a pyrophosphoric acid solution. Next, the above-mentioned iron solution was gradually added to the pyrophosphoric acid solution with stirring, and the pH of the mixture was adjusted to 3.0. The salt-formation of ferric pyrophosphate due to neutralization was terminated, and thereafter the mixture was subjected to a liquid-solid separation by centrifugation (3000G, 5 minutes), to give an emulsifying agent-coated iron salt composition A in a solid phase

portion. Subsequently, the resulting composition was dispersed by adding 800 ml of ion-exchanged water, to give 860 ml of a solution of an emulsifying agent-coated iron salt composition A.

The average particle size of this composition as determined by laser diffraction particle size distribution was 0.2 μm, and the iron content was 1.2% by weight.

Preparation of Enriched rice

Enriched Rice 1

In a coating pan, 2 kg of rice was immersed in 400 ml of a 9% aqueous acetic acid solution containing 9.0 g of dibenzoylthiamine hydrochloride, 0.2 g of vitamin B_2, 15.0 g of nicotinamide, 9.0 g of calcium pantothenate and 2.0 g of pyridoxine hydrochloride at a bath temperature of about 35°C for 2 hours. The immersed rice was taken out and steamed in a water vapor at about 100°C for about 2 minutes and finally dried in a warm air current of about 70°C for about 1 hour. Then, the rice was sieved to remove bound and crashed grains to thereby give 1.94 kg of dried rice with a water content of 12.6%. The rice was transferred to the coating pan, where it was spray-coated with 250 g of an aqueous suspension containing 13 g of natural vitamin E, 50 g of calcium carbonate, 10 g of sucrose and 10 g of α-starch. Then, this was spray-coated with 100 ml of an aqueous suspension containing 15 g of ferric pyrophosphate, 15 g of sucrose and 5 g of gum arabic. The rice was further spray-coated with 500 g of an emulsion containing 100 g of hydrogenated cottonseed oil, 5 g of rice wax, 10 g of sucrose fatty acid ester (HLB 2) and 385 g of water. The above enriched polished rice was polished by tumbling it in a coating pan at an internal temperature of about 55°C until the grains presented glossy surfaces. Then, the rice was spray-coated with a

solution of 2 g of sucrose fatty acid ester (HLB 15) in 100 ml of water and further with 300 g of an aqueous suspension containing 100 g of wheat starch, 40 g of sugar, 4 g of α-starch and 0.1 g of vitamin B_2 to give about 2.1 kg of Enriched Rice 1.

Enriched Rice 2

In a coating pan, 2 kg of rice was immersed in 400 ml of a 9% aqueous acetic acid solution containing 9.0 g of dibenzoylthiamine hydrochloride, 0.2 g of vitamin B_2, 15.0 g of nicotinamide, 9.0 g of calcium pantothenate and 2.0 g of pyridoxine hydrochloride at a bath temperature of about 35°C for 2 hours. The immersed rice was taken out and steamed in a water vapor at about 100°C for about 2 minutes and finally dried in a warm air current of about 70°C for about 1 hour. Then, the rice was sieved to remove bound and crashed grains to thereby give 1.94 kg of dried rice with a water content of 12.6%. The rice was transferred to the coating pan, where it was spray-coated with 250 g of an aqueous suspension containing 13 g of natural vitamin E, 50 g of calcium carbonate, 10 g of sucrose and 10 g of α-starch. Then, this was spray-coated with 300 ml of the emulsifying agent-coated iron salt composition A, 15 g of sucrose and 5 g of gum arabic. The rice was further spray-coated with 500 g of an emulsion containing 100 g of hydrogenated cottonseed oil, 5 g of rice wax, 10 g of sucrose fatty acid ester (HLB 2) and 385 g of water. The above enriched polished rice was polished by tumbling it in a coating pan at an internal temperature of about 55°C until the grains presented glossy surfaces. Then, the rice was spray-coated with a solution of 2 g of sucrose fatty acid ester (HLB 15) in 100 ml of water and further with 300 g of an aqueous suspension containing 100 g of wheat starch, 40 g of sugar, 4 g of α-starch and 0.1 g of vitamin B_2 to give about 2.1 kg of Enriched Rice 2.

Enriched Rice 3

9.0 g of dibenzoylthiamine hydrochloride, 0.2 g of vitamin B_2, 15.0 g of nicotinamide, 9.0 g of calcium pantothenate and 2.0 g of pyridoxine hydrochloride were mixed to prepare a vitamin premix. 13g of natural vitamin E and 2.6 g of a polyglycerol fatty acid ester (SUNSOFT AZ18G: manufactured by Taiyo Kagaku Co., Ltd.) were dissolved in 36.4 ml of deionized water, to prepare vitamin E emulsion with a homomixer. To 300 ml of an aqueous suspension containing 15 g of ferric pyrophosphate were added an entire amount of the above-mentioned vitamin premix, an entire amount of the above-mentioned vitamin E emulsion and 50 g of calcium carbonate, and the mixture was stirred to give a dispersion of minerals and vitamins.

The amount 2.0 kg of polished rice was placed in a coating pan, and an entire amount of the above-mentioned dispersion was sprayed thereto at a rate of 5 ml/minute, while rotating the coating pan, and blowing hot air thereinto, to coat the polished rice. Hot air was continued to be blown for dryness even after the termination of spraying of the dispersion. A portion having fine sizes was removed with an 8-mesh sieve, to give 2.0 kg of iron salt- and vitamin-coated rice having a water content of 10%.

19 g of cottonseed hydrogenated oil, 1 g of hexaglycerol octastearate (SUNFAT PS68: manufactured by Taiyo Kagaku Co., Ltd.) and 0.5 g of hexaglycerol condensed ricinoleate (SUNSOFT 818H: manufactured by Taiyo Kagaku Co., Ltd,) were dissolved and mixed at 80°C for 10 minutes, to prepare a fat or oil for coating. Next, 500g of the above-mentioned iron salt- and vitamin-coated rice was placed in a coating pan. While rotating the coating pan, a wind normal temperature was blown, and the above-mentioned fat or oil for coating was sprayed at a rate of 2.5 g/minute, while keeping the

temperature of the above-mentioned fat or oil for coating at 60°C, to give 520 g of Enriched Rice 3.

Enriched Rice 4

9.0 g of dibenzoylthiamine hydrochloride, 0.2 g of vitamin B_2, 15.0 g of nicotinamide, 9.0 g of calcium pantothenate and 2.0 g of pyridoxine hydrochloride were mixed to prepare a vitamin premix. 13g of natural vitamin E and 2.6 g of a polyglycerol fatty acid ester (SUNSOFT AZ18G: manufactured by Taiyo Kagaku Co., Ltd.) were dissolved in 36.4 ml of deionized water, to prepare vitamin E emulsion with a homomixer. To 300 ml of the emulsifying agent-coated iron salt composition A were added an entire amount of the above-mentioned vitamin premix, an entire amount of the above-mentioned vitamin E emulsion and 50 g of calcium carbonate, and the mixture was stirred to give a dispersion of minerals and vitamins.

The amount 2.0 kg of polished rice was placed in a coating pan, and an entire amount of the above-mentioned dispersion was sprayed thereto at a rate of 5 ml/minute, while rotating the coating pan, and blowing hot air thereinto, to coat the polished rice. Hot air was continued to be blown for dryness even after the termination of spraying of the dispersion. A portion having fine sizes was removed with an 8-mesh sieve, to give 2.0 kg of iron salt- and vitamin-coated rice having a water content of 10%.

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rotating the coating pan, a wind normal temperature was blown, and the above-mentioned fat or oil for coating was sprayed at a rate of 2.5 g/minute, while keeping the temperature of the above-mentioned fat or oil for coating at 60°C, to give 520 g of Enriched Rice 4.

Test Methods

1. Residual Ratio Test of Vitamins and Minerals in Enriched Rice

The residual ratios of vitamins and minerals in the finally prepared enriched rice to the theoretical contents of vitamins and minerals were obtained from the amounts of vitamins and minerals used. The results are summarized in Table I. Here, the vitamin content and the mineral content were measured in according to the basis of Standard Methods of Analysis for Hygiene Chemists in Japan.

Table I

Item	Residual Ratio (%) in Final Nutrition Enriched Rice			
	Enriched Rice 1	Enriched Rice 2	Enriched Rice 3	Enriched Rice 4
Vitamin B_1	29.8	69.0	35.4	78.9
Vitamin B_2	23.3	20.0	70.0	73.3
Vitamin B_6	7.3	9.1	53.5	63.3
Vitamin E	47.5	54.7	69.0	80.7
Iron	50.5	62.4	86.0	91.6

Misaki involves a complicated production method, requiring a larger number of spray-coating steps, so that the residual ratios of vitamins and minerals are undesirably low. In addition, as is clear from the comparisons between Enriched Rice 1 and Enriched Rice 2 and between Enriched Rice 3 and Enriched Rice 4, the enriched rice

prepared using the emulsifying agent-coated iron salt composition A also has an excellent residual ratio for vitamins.

2. Run-off Test of Vitamins and Minerals Upon Washing Rice

1 g of iron- and vitamin-enriched rice was respectively mixed with 200 g of polished rice, to give test sample rice. The procedures of adding 250 ml of tap water to each of test sample rice, washing the test sample rice with water so as to rub and loosen at a rate of about 30 rotations per minute, and draining water therefrom were repeated four times. The drained water was collected, and the amounts of iron and vitamins contained in the drained water were determined. The percent loss was obtained by calculating a ratio of the content to that in 1 g of iron- and vitamin-enriched rice.

Here, the amounts of iron and vitamins were determined according to the basis of Standard Methods of Analysis for Hygiene Chemists in Japan.

Table II

Item	Loss Percentage (%) by Washing Rice			
	Enriched Rice 1	Enriched Rice 2	Enriched Rice 3	Enriched Rice 4
Vitamin B_1	2.31	1.30	4.27	3.87
Vitamin B_2	4.91	3.78	4.90	4.18
Vitamin B_6	4.67	4.54	4.23	1.64
Vitamin E	3.93	1.81	2.57	2.59
Iron	3.97	3.49	2.85	2.18

As is clear from the comparisons between Enriched Rice 1 and Enriched Rice 2 and between Enriched Rice 3 and Enriched Rice 4, it is found that the loss percentage is

likely to be lowered by washing rice when the emulsifying agent-coated iron salt composition A is used.

3. Test for Residual Vitamins and Minerals upon Storage

The iron- and vitamin-enriched rice was stored at room temperature for one month, and thereafter the contents of vitamin B_1, Vitamin B_2, Vitamin B_6, Vitamin E and iron were determined in each of iron- and vitamin-enriched rice. The residual ratio was obtained by calculating a ratio of the content to that before storage. Here, the amounts of iron and vitamins were determined according to the basis of Standard Methods of Analysis for Hygiene Chemists in Japan.

Table III

Item	Residual Ratio (%) After One-Month Storage			
	Enriched Rice 1	Enriched Rice 2	Enriched Rice 3	Enriched Rice 4
Vitamin B_1	51.6	84.3	56.1	85.4
Vitamin B_2	85.9	94.3	88.6	93.7
Vitamin B_6	61.7	79.8	59.3	81.4
Vitamin E	54.8	66.2	58.7	63.4
Iron	103.6	101.2	99.7	100.7

As is clear from the comparisons between Enriched Rice 1 and Enriched Rice 2 and between Enriched Rice 3 and Enriched Rice 4, the enriched rice using the emulsifying agent-coated iron salt composition A has excellent storage stability.

Statement Under 18 U.S.C. § 1001

The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Noboru Sakaguchi

Noboru SAKAGUCHI

August 27, 2010

Date